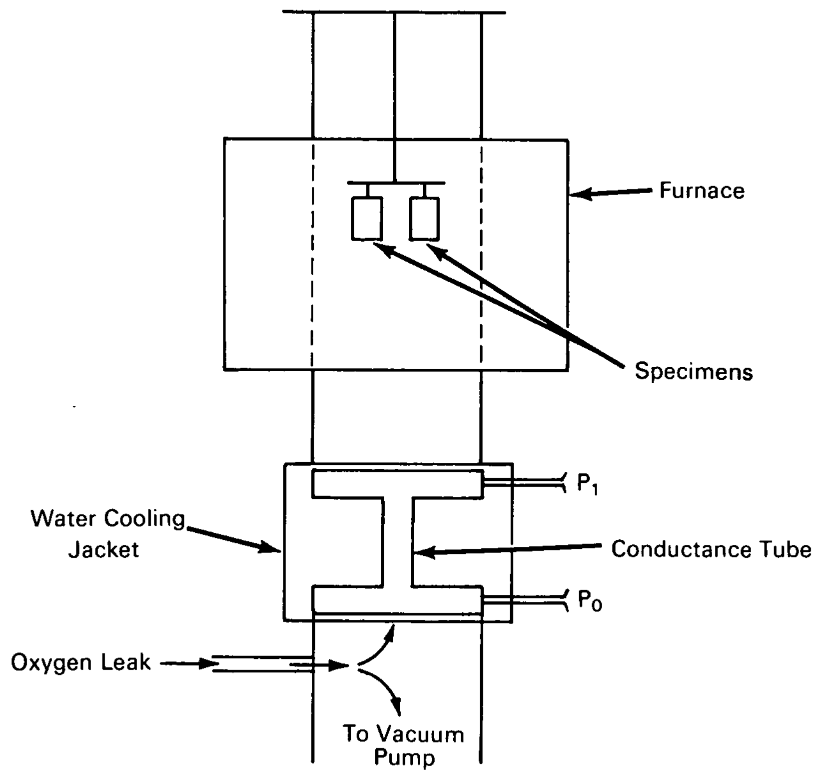
NASA TECH BRIEF

Precise Doping of Metals by Small Gas Flows



Doping of refractory metal specimens with controlled additions of oxygen is used to study the effects of oxygen on various properties of the specimens, such as alkali metal-corrosion-resistance and creep. Methods previously used to control oxygen addition are not entirely satisfactory. The use of a carrier gas (e.g., argon) with small concentrations of oxygen, generally requires large amounts of gas and presents the problem of uniform gas distribution among specimens. Volumetric methods require precise temperature control.

In a relatively simple method which was developed for doping with oxygen, the metal specimens are heated in a dynamic high-vacuum system (see schematic). A controlled oxygen in-leak is provided, and the specimens absorb the incoming oxygen. In principle, it is only necessary to meter the amount of gas being absorbed by the specimens to control the process in a reproducible manner. To accomplish this, the oxygen leak rate is adjusted by trial and error so that a required pressure is maintained at a given temperature for a specified time.

(continued overleaf)

The oxygen leak is supplied between the vacuum pump and the specimen chamber so that the gas flow is divided between them. Part of the gas is then carried away by the vacuum pump and part flows into the chamber through a flow conductance tube. Gas flow into the chamber is measured by the difference in pressure ($P_0 - P_1$) sensed by thoriated-iridium ionization gages tapped into each end of the conductance tube. The rate of oxygen absorption, R, by the specimens, in grams per hour, is calculated from the following formula:

$$R = 57.729 \, (P_0 - P_1) \, C_0 \frac{M}{T},$$

where C_0 is the flow conductance, M is the molecular weight of the gas and T is the temperature of the conductance tube.

The conductance is sized so that for a desired flow rate, the pressure differential ($P_0 - P_1$) is sufficiently large to preclude the introduction of appreciable errors. If the conductance is too small, however, the back pressure (at the point where P_1 is measured) will be too large. The pressure (ionization) gages are calibrated using oxygen and remain drift-free; the only variable is the temperature of the conductance tube. This tube can be cooled to keep its temperature essentially constant. The precision of the absorption measurement in a given run is approximately 3 percent; measurement variability between like specimens suspended symmetrically at the same level is approximately 2 percent.

Notes:

1. An automatic readout of oxygen absorption can be obtained with an electronic integrating device.
2. The system can be used for other oxygen absorption processes (such as low-pressure oxidation measurements) and for gases other than oxygen.
3. For small (laboratory-size) specimens, only a few hours are required to complete an oxygen doping run. Less than 1 percent of the gas volume of a standard oxygen bottle is consumed.
4. Documentation is available from:

   Clearinghouse for Federal Scientific and Technical Information
   Springfield, Virginia 22151
   Price $3.00
   Reference: B68-10526

Questions concerning this innovation may also be directed to:

Technology Utilization Officer
Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Reference: B68-10526

Patent status:

No patent action is contemplated by NASA.

Source: C. A. Barrett
(LEW-10444)